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SOURCING OBSIDIAN BY SEM-EDS AND EDXRF IN NEOLITHIC SYRIA

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In this study **[1]**, we used two non-destructive techniques, scanning electron microscopy with an energy dispersive spectrometer (SEM-EDS; **[2]**) and energy dispersive x-ray fluorescence (EDXRF; **[2 ; 3]**), for the elemental characterisation of obsidian assemblages from Tell Aswad (middle/late Pre-Pottery Neolithic B, PPNB; **[4]**) and Qdeir 1 (final PPNB; **[5]**), two Neolithic sites in Syria **[6]**. Firstly, we show for the first time the efficiency of SEM-EDS in discriminating the eastern Anatolian peralkaline sources of Bingöl A and Nemrut Dağ, two of the most important sources in Near Eastern prehistory, often difficult to differentiate due to their geochemical similarity. Secondly, the rapidity of EDXRF **[7 ; 8]** allowed us to analyse the entire assemblages, revealing new data on the obsidian provenance of the two sites.

SEM-EDS analysis were conducted at the CRP2A (Bordeaux, France) using a JEOL JMS 6460 LV scanning electron microscope. The study involved 61 artefacts from Tell Aswad and 180 from Qdeir 1, *i.e.* 58% and 35% of our assemblages. Na, Mg, Al, Si, K, Ca, Ti, Mn and Fe contents were obtained for each piece following the procedure of Le Bourdonnec *et al.* (2010 ; **[12]**). Due to the artefacts' sizes and geometry, the elemental composition was determined as the average of two to eight 'punctual' measurements.

EDXRF analysis were undertaken in the McMaster Archaeological XRF Lab [MAX Lab] using a Thermo Scientific Quant'X energy dispersive x-ray fluorescence spectrometer; the protocols and methods following those of Shackley (2005 ; **[13]**). Fifteen major and trace elements were recorded: Ti, Mn, Fe, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, Pb and Th. Very small artefacts, or those exhibiting anomalous concentrations, were re-run to ensure accuracy and precision.The speed and automation of this technique (approximately 3.5 hours for 19 artefacts plus the standard) enabled us to analyse the whole assemblages.

Tell Aswad is a PPNB site in southern Syria (Damascus basin), excavated by the El Kowm-Mureybet permanent mission of the Maison de l'Orient et de la Méditerranée (Lyon, France) since 1972. The chronology of the site, established by C^{14} dating in 2003 **[9]**, dates back to the '*Néolithique Ancien de Byblos*', which spans from 8654-8319 cal BC to 7730-7551 cal BC. The obsidian assemblage detailed here came from the 2001, 2002 and 2003 excavations and were entrusted to us by Frédéric Abbès (MOM). All the pieces come from the middle/late PPNB levels (c. 8200-7500 cal BC; **[4]**).

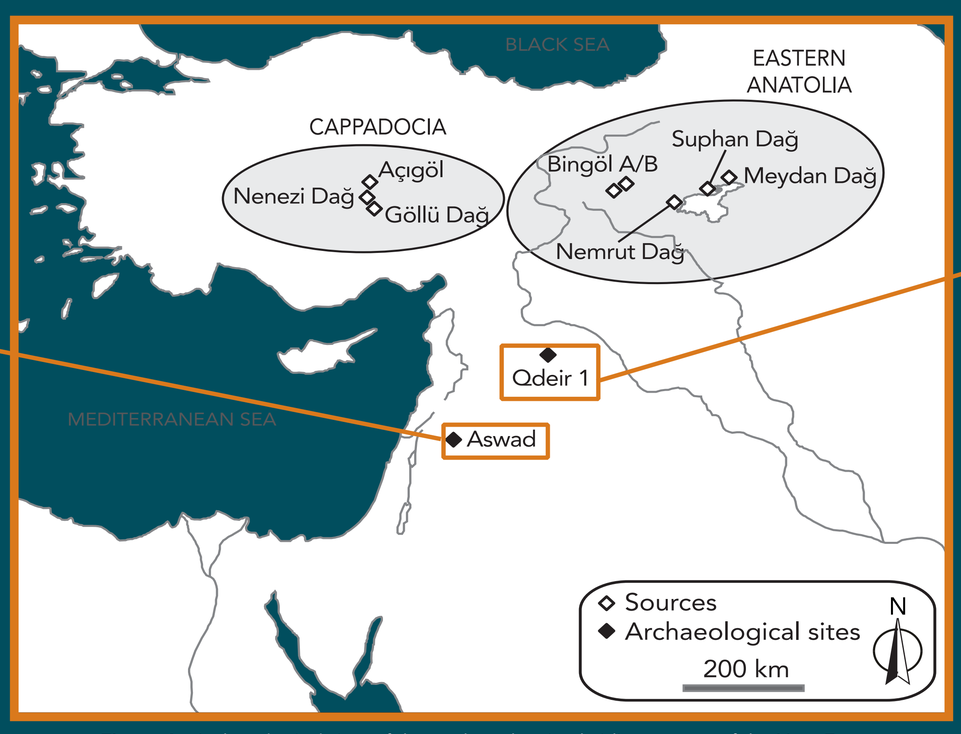
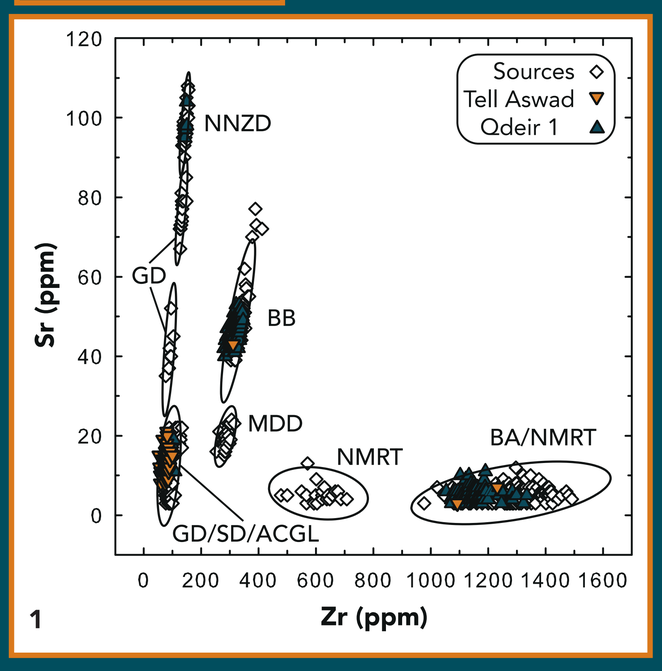
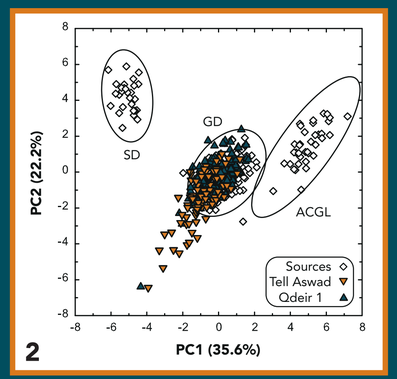


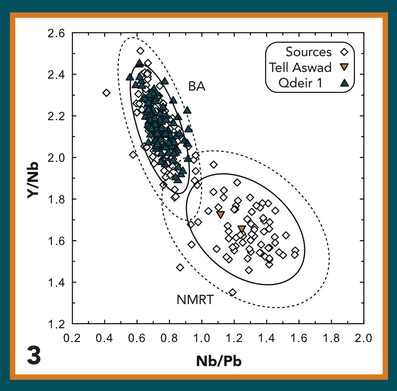
Figure 1 : Archaeological sites of the study and main obsidian sources of the Near East

Qdeir 1 was first surveyed by Olivier Aurenche in 1980, revealing a final PPNB settlement (7100-5720 cal BC) **[10]**. It was then excavated as a part of the El-Kowm-Mureybet permanent mission (MOM). The site, only 8 kilometers distant from the El Kowm village **[11]** (first surveyed in 1967), has a long and complex settlement sequence (between 7100 and 4230 cal BC **[10]**, with at least five different levels of occupation. The archaeology of Qdeir 1 comprises a few structures, a semi-permanent camp, and a flint and obsidian workshop. Its characteristics lead Qdeir 1 to be attributed to the «*PPNB du désert*». The obsidian from all Qdeir 1 contexts is present in the form of pressure blades and bladelets made of various raw materials **[10]**.

EDXRF Analysis



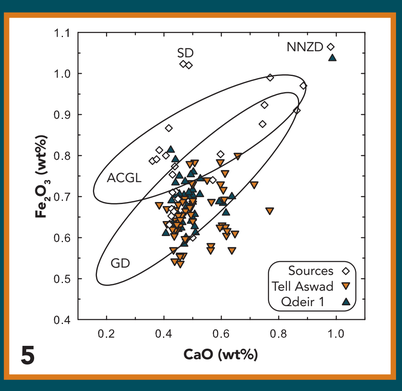


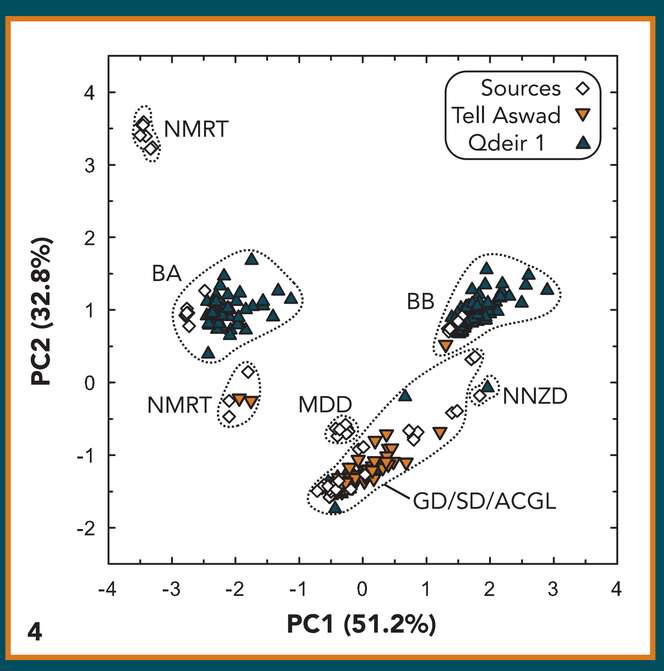


Graph 1: Zr vs. Sr contents determined by EDXRF for Tell Aswad and Qdeir 1 artefacts plus source samples; 99% normal density ellipses.
Graph 2: Principal components analysis using Ti, Mn, Fe, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, Pb and Th contents obtained by EDXRF, comparing obsidians from Tell Aswad, Qdeir 1 and source samples from Göllü Dağ, Suphan Dağ and Acıgöl. 99% normal density ellipses.
Graph 3: EDXRF ratio plot of Nb/Pb vs. Y/Nb for Tell Aswad, Qdeir 1 and source samples from Bingöl A and Nemrut Dağ. 99% (dotted line) and 90% (solid line) normal density ellipses.
Source abreviations: BA (Bingöl A), BB (Bingöl B), ACGL (Acıgöl), GD (Göllü Dağ), SD (Suphan Dağ), NNZD (Nenezı Dağ), NMRT (Nemrut Dağ), MDD (Meydan Dağ).

SEM-EDS Analysis

Graph 4: Principal components analysis (PCA) using Al, Si, Ca and Fe contents determined by SEM-EDS, comparing obsidians from Tell Aswad, Qdeir 1 and source samples.
Graph 5: CaO vs. Fe2O3 contents determined by SEM-EDS for Tell Aswad and Qdeir 1 artefacts plus source samples from Göllü Dağ, Suphan Dağ, Nenezi Dağ and Acıgöl. 90% normal density ellipses.





	TELL ASWAD		QDEIR 1	
	SEM-EDS	EDXRF	SEM-EDS	EDXRF
Göllü Dağ	58	100	40	143
Nenezı Dağ	-	-	1	4
Bingöl B	1	1	89	230
Bïngol A	-	-	47	102
Nemrut Dağ	2	2	-	-
Total artefacts analysed	*61*	*103*	*177*	*479*
Total artefacts assemblage	*105*		*517*	

Table 1: Total number of Tell Aswad and Qdeir 1 obsidian artefacts analysed by SEM-EDS and EDXRF.

The EDXRF analysis allowed us to assign Qdeir's aretfacts to four different obsidian sources (**Graphs 1, 2 and 3**; **Table 1**): Bingöl B, Bingöl A, Göllü Dağ and Nenezı Dağ. At Tell Aswad, only three sources were represented: Göllü Dağ, Nemrut Dağ and Bingöl B. The discrimination between the Cappadocian sources of Acıgöl and Göllü Dağ was clarified by a Principal Component Analysis (see **Graph 2**). Regarding the peralcaline products, all of them were assigned to Bingöl A by a Nb/Pb vs. Y/Nb ration diagram.

Four different raw materials were also determined by SEM-EDS for both assemblages. However, this method does not allow us to distinguish between the calco-alkaline products of Acıgöl and Göllü Dağ **[2]**, but we show for the first time that it can make the distinction between the peralcaline products of Nemrut Dağ and Bingöl A. Indeed, all Qdeir's aretfacts were successfully attributed to the Bingöl A source, and those of Tell Aswad to the Nemrut Dağ source, as with the EDXRF analyses.

Together with the technological study of both assemblages, the EDXRF and SEM-EDS analyses allowed us to make some conclusions on the obsidian economy of Tell Aswad and Qdeir 1.

At Tell Aswad:

- the use of Bingöl B obsidian during middle/late PPNB
- a preferential use of Nemrut Dağ peralkaline products over those of Bingöl A
- different ***chaînes opératoires*** and an important 'recycling' rate of debitage, often through the use of the burin blow technique ;

At Qdeir 1:

- the use of a South Cappadocian obsidian source (Göllü Dağ)
- a preferential use of Bingöl A peralkaline products over those of Nemrut (Göllü Dağ)
- several chaînes opératoires (unipolar and bipolar debitage, and use of the pressure technique for some unipolar blades).

The authors would like to thank Frédéric Abbès for providing the obsidian assemblage from Tell Aswad and for the help in the study of the chaînes opératoires. We also thank Pierre Machut and Brigitte Spiteri for the help during sample preparation for SEM-EDS analysis. The MAX Lab was established and is currently operated by a Canada Foundation for Innovation Leader's Opportunity Fund / Ontario Research Fund. Orange's time at the MAX Lab was funded by Aquimob (mobility grant allocated by the Region Aquitaine). This project was partly coordinated by the University of Bordeaux and has received support from the National Agency for Research under the 'future investment' program (ANR-10-52-LabX) while Carter's involvement was covered by a Standard Research Grant of the Social Sciences and Humanities. Research Council, Canada.

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